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SOME FACTORS AFFECTING THE RATE OF VULCANIZATION OF PLANTATION RUBBER.

By R. O. Bishop.

Introduction.

As a result of the experimental work carried out by Eaton, Whitby, Stevens, De Vries and others it has been established that the optimum rate of vulcanization of plantation rubber is dependent upon the method by which the raw rubber is prepared from its parent latex; and within recognised limits the intrinsic vulcanizing properties of a piece of raw rubber can therefore be controlled by the producer.

The rubber which is produced at the present as plantation standard rubber may be divided into three groups according to the rate of vulcanization determined under standard conditions of a simple 9 ; 1 rubber sulphur mix. Thus 1st latex crepe has an optimum rate of vulcanization of approximately 3 hours, smoked sheet approximately 2½ hours, matured rubber or "slab" approximately 60 minutes.

It is not proposed to discuss whether these differences are of fundamental importance to the value of the rubber as judged by the manufacturer of vulcanized material or not, but, accepting the fact that the differences exist, it appears to be supremely important to obtain all the information which can be derived from a study of these differences in the rates of vulcanization of raw rubber.

Referring to work by previous investigators such as Whitby, Stevens, De Vries and Eaton it may be recalled that the most striking variations in rate of vulcanization of plantation rubber have been attributed to the incorporation with the caoutchouc of varying amounts of nitrogenous non-caoutchouc material which functions as an accelerator of the rubber sulphur combination.

Eaton, Day and Grantham[1] record work from which they infer that the nitrogenous accelerator exists in two forms or that there are two accelerators, one pre-formed in the latex, and of which a small proportion only is present normally in raw rubber prepared by

ordinary coagulation methods, while the other is formed from a non-caoutchouc protein material by decomposition brought about by certain organisms which are supposed to function under anaerobic conditions.

This latter accelerating substance appears to have received the most attention from investigators. It has been stated that the protein of the latex is decomposed into amino bodies during the maturation period and that sufficient of these bodies remain with the caoutchouc after washing and subsequent drying to cause acceleration.

The work of Stevens[2] carried out with the object of isolating such nitrogenous accelerators did not tend to confirm these views. Stevens extracted matured rubber with water for several days, precipitated the protein in the resulting solution by tannic acid, removed the tannic acid by barium hydroxide and then added phosphotungstic acid to the solution. By such means the phosphotungstate of a base was obtained, the total amount of the compound being approximately 0.03% on the original rubber. The base was not identified but it was assumed that the same substance could be isolated from serum.

Larger quantities were prepared from the serum (presumably "matured") which separated in the coagulating pans from 10 litres of latex, equivalent to 1,500 grms. rubber at the least. The amount was 0.59 grms. of accelerator which is significant in view of the fact that from a subsequent comparison of the accelerating properties of this substance with artificial accelerators it was concluded that the "base" isolated above was somewhat less powerful than p-nitrosodimethylaniline.

The latest contribution to our knowledge on the subject is contained in the work recently carried out by De Vries* who has drawn noteworthy comparisons between the rates of vulcanization of whole rubber and matured rubber. Certain details of the results recorded by De Vries are not in agreement with those to be reported here and it hoped that a statement of these differences will serve still further to illuminate the problem of latex composition and its influence on the vulcanizing properties of the derived rubber.

Experimental.

Alcohol Coagulation.—If fresh latex is dropped into 95% alcohol coagulation takes place instantaneously. The semi-granular coagulum can be collected without causing the granules to cohere, so that effective washing can afterwards be carried out. The washed mass can be filtered and partially dried on a Buchner funnel.

The coagulum can then be pressed in a hand-press and subsequently creped to a thin sheet which will dry completely in a few hours at the laboratory temperature. Should drying not be desired

* Archief voor de Rubberculture. August 1923.

the washed coagulum can be squeezed into a ball by hand and left for 7 days so that any "maturation" possible under the circumstances may take place.

By adopting this procedure some interesting differences were established in the rate of vulcanization of rubber precipitated from latex by alcohol. To illustrate these differences samples are described which were prepared in experiment 222.

A. 1000 c.c. of latex dropped into 3000 c.c. of alcohol which had been acidified by the addition of N/10 H_2SO_4 to adjust the pH value to 3. The temperature being that of the laboratory, viz: 25°—30°C throughout, and N/10 H_2SO_4 being added from time to time to maintain the pH value constant.

B. 1000 c.c. of the same latex dropped into 3000 c.c. of alcohol maintained at the same constant pH value (approximately 3), but the operation conducted at a temperature of 60°C.

C. 1000 c.c. of the same latex dropped into 3000 c.c. of alcohol of which the pH value was adjusted and maintained throughout at approximately 7. Temperature being about 25°C.

D. 1000 c.c. of the same latex dropped into 3000 c.c. of alcohol with pH value maintained at approximately 7 as for C above but having the temperature constant at 60°C.

E. 1000 c.c. of the same latex dropped into 3000 c.c. of alcohol of which the pH value was adjusted and maintained throughout at approximately 9. Temperature being 25°C.

F. 1000 c.c. of the same latex dropped into 3000 c.c. of alcohol as for E. above with pH value maintained at approximately 7 but having the temperature constant at 60°C.

The coagula obtained were washed and freed from adhering liquid. They were divided into equal portions—500 c.c. of latex providing one portion and the other 500 c.c. of latex providing the second portion. From each type of preparation one portion of coagulum was squeezed into a lump and kept 7 days, the other portion of coagulum was pressed into a sheet, creped, and dried within a few hours. After the lapse of 7 days the "matured" coagula were pressed, creped, and dried in a manner exactly similar to the corresponding portion previously creped.

The following table gives the results obtained by vulcanizing the rubber under uniform conditions in a 9 : 1 rubber sulphur mix.*

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* For particulars of vulcanization refer Department of Agriculture Bulletin No. 27.

TABLE I.

Samples.	Temperature of coagulation.	pH of Alcohol. (approx.)	Optimum time of vulcanization of crepe. (minutes).	Optimum time of vulcanization of matured rubber. (minutes).
222 A.	25.°C.	3	150	135
222 B.	60.°C.	3	120	120
222 C.	25.°C.	7	150	135
222 D.	60.°C.	7	105	105
222 E.	25.°C.	9	105	70
222 F.	60.°C.	9	50	50

Matured slab rubber coagulated from the same latex in the standard manner by adding 3 ozs. of a 5% acetic acid solution to a gallon of latex, showed an optimum rate of vulcanization of 65 minutes.

Crepe rubber prepared in the standard manner by coagulating with acetic acid showed an optimum rate of vulcanization of 150 minutes.

From these figures the following facts appear :—

1. There is a striking progressive acceleration in vulcanization from samples A. B. to samples E. F.

2. The use of hot instead of cold alcohol for coagulation causes an accelerated rate of vulcanization of the rubber obtained.

3. The acceleration of vulcanization caused by the cold "alkaline" coagulation is equal to the acceleration of vulcanization caused by the hot neutral alcohol compared with the cold neutral alcohol. The later is equal to the control.

4. The coagula derived from cold alcohol coagulations will, on keeping, develop and accelerated rate of vulcanization while no change occurs on keeping the coagula from hot alcohols.

Possible explanations of these facts may be enumerated as follows :—

A. Chemical. The added acid or alkali exerts an influence on the rubber by virtue of its intrinsic chemical properties.

B. Physical. The variations in hydrogen ion concentration of the latex owing to the added acid or alkali may cause physical changes which will result in alterations in the properties of the coagulated rubber.

C. The non-caoutchouc constituents may account for the variations in the rubber by their different solubilities in the "acid" and "alkaline" media.

D. There may be a combination of two or more of these explanations.

The possibilities will be examined under each heading.

A. It is relevant to record the amounts of acid and alkali which were used to produce the hydrogen ion concentrations in the three series of coagulations. In the "alkaline" coagulations the quantity of H_2SO_4 used was approximately 0.06% by weight of the total weight of latex plus alcohol for coagulation. In the "alkaline" coagulations the quantity of NaOH used was approximately 0.07% by weight of the weight of latex and alcohol. These amounts are of the same order, and, for the acid within the limits prescribed for ordinary standard acid coagulation on a modern estate.

The following lines of action were adopted for further investigation :—

(i) A common stock of latex was coagulated as before by dropping into alcohol at 60°C. The hydrogen ion concentration of the alcohol being adjusted so that in one case pH was maintained at *c*3. and in another case at *c*9. A second "acid" coagulation and a second "alkaline" coagulation was then made with the same latex but the quantity of N/10 H_2SO_4 and N/10 Na OH added to the alcohol was doubled at each addition—the pH value of the alcohol being disregarded. By this means it was supposed that any absorption of mineral acid or alkali by the rubber during coagulation would be reflected in the optimum time of vulcanization of the dry crepe prepared from the coagula thrown down under extremes of "acid" and "alkali."

(ii) The samples of crepe prepared during the previous coagulations and under (*a*) above were to be examined for relative alkalinity or acidity of ash. For this purpose 5 grms. of the sample were ashed at the lowest possible temperature consistent with complete combustion of the carbon. The total ash was then digested with 100 c.c. water for 10 mins. and the pH value of the filtrate determined under uniform conditions.

In connection with this work it was fully realised that uncertainty must prevail as to the reactions occurring during the incineration of such a body as rubber, and the consequent combinations in which the mineral matter would appear in the final ash. Therefore it was felt that digesting the ash with water for a pH determination was open to criticism. And it was decided that no deductions could be drawn from negative results, although, it might be possible to establish sufficiently large relative differences between the "acid" and "alkaline" rubber ashes to indicate the lines for further investigation.

(iii) A third method adopted was the examination of the relative "acidity" of emulsions prepared from the dry crepe samples under constant conditions.

One gramme of the crepe was dissolved in 100 c.c. of benzene or petrol, and the solution kept in the dark for 4 days until homogenous. Five c.c. were then removed, shaken with an equal volume of alcohol and to the emulsion was then added 1 c.c. of water. The separated water was examined by the use of a range of indicators until the approximate pH was registered by comparison with a standard.

Table II gives the results obtained under the 3 methods (*a*) (*b*) and (*c*) above.

The figures show the inadequacy of the explanation that it is the chemical presence of the mineral acid or alkali which is the cause of the variation in the vulcanizing properties of the series of samples prepared in experiment 222 and previously described. They are also interesting as additional proofs of the natural variations which occur in latex.

Elimination of the obvious chemical explanation consequently forces one to consider the possible explanation based on physical grounds. It is apparent from an examination of the benzene emulsions, if reliance can be placed on the degree of difference indicated by the pH determinations, the a very small alteration in the pH value of a rubber will correspond to a considerable variation in rates of vulcanization.

TABLE II.

Rubber Ref. No.	Coagulant.	Optimum rate of vulcanization (minutes.)	Ash per cent.	pH Value of Ash extract approx.	pH Value of Benzene emulsion approx.	pH Value of Petrol emulsion approx.
222 K.	"Acid"	155	—	8	6	7
222 L.	Double "Acid"	150	0.36	8	6	7
222 M.	"Alkali"	75	0.29	7	7	8
222 N.	Double "Alkali"	75	0.49	9	7	8
222 O.	"Acid"	160	0.27	7	6	—
222 P.	"Alkali"	100	0.31	9	6	—
222 T.	Control Spontaneous coagulation*	105	0.37	10	6	—
234 HAH.	Special †	35	0.49	8	7	—
236 HAK.	Special †	40	0.30	8	—	—

* The sample 222 T. was a piece of crepe prepared from the coagulum obtained by allowing a quantity of the same stock to coagulate spontaneously without the addition of any coagulant and then maturing the coagulum for 7 days.

† These samples will be described fully on page 324. The results are inserted in this table for the purpose of comparing a rubber with an exceptionally rapid rate of vulcanisation obtained without an alkaline coagulant.

Experiment 235 was made to investigate the significance of small differences in hydrogen ion concentration of latex at the time of coagulation. In this experiment two series of coagulations were carried out on a common stock of latex. Four coagulations in the first series were designed to give samples of crepe, 235A—235D, from the same dilutions of latex, coagulated with a constant volume concentration of acetic acid, and necessarily under varying pH values. The four coagulations in the second series were designed to give samples of crepe, 235Ax—235Dx, from different dilutions of latex, with the additions of such proportions of acetic acid that the pH value of the latex at coagulation was constant, the constant value being the normal obtained when acetic acid is used as a coagulant under normal conditions. The coagula obtained at the end of 24 hours, were all washed, creped immediately and dried in a uniform manner. A description of the samples is summarised below :—

235A. Latex of 36% dry rubber content coagulated at pH 5.8 Coagulation was imperfect after 24 hours. The coagulum showed brown patches. The serum was milky and had a pH value of 4.4.

235B. Latex of 27% dry rubber content coagulated at pH 5.6 Coagulation less imperfect than A. The coagulum was pinkish. The serum was less milky than A. Serum pH 4.3.

235C. Latex of 15% dry rubber content coagulated at pH 5.4 Coagulation was perfect. The serum was colourless and clear and the coagulum was white. Serum pH 4.3.

235D. Latex of 10% dry rubber content coagulated at pH 5.2 Coagulation equally perfect with C. Serum pH 4.3.

235Ax. Latex of 36% dry rubber content coagulated at pH 5.4 Coagulation was imperfect after 24 hours. The coagulum showed brown patches. The serum was milky and had a pH value 4.3.

235Bx. Latex of 27% dry rubber content coagulated at pH 5.4 Coaguiation less imperfect than Ax. Coagulum pink. Serum milky, had a pH 4.3.

235Cx. Latex of 15% dry rubber content, coagulated at pH 5.4 Coagulation perfect, coagulum white, serum colourless and had pH 4.3.

235Dx. Latex 10% dry rubber content, coagulated at pH 5.4 Coagulation not so perfect as Cx. Serum somewhat milky, pH 4.3.

The samples of crepe were vulcanized side by side under the usual conditions adopted throughout this work. The serum in each case was examined for total solids by pipetting out an aliquot portion

and evaporating to dryness on the water bath. The following results were obtained :—

TABLE III.

Sample.	pH. at coagulation.	Optimum rate of vulcanization (minutes.)	Percentage Total solids in Serum Liquid.
235A.	5.8	135	6.4
235B.	5.6	150	3.3
235C.	5.4	180	3.0
235D.	5.2	180	1.8
235Ax.	5.4	135	6.8
235Bx.	5.4	165	3.3
235Cx.	5.4	180	3.2
235Dx.	5.4	180	2.4

The figures show that relatively small differences in hydrogen ion concentration of the latex are without appreciable effect on the rate of vulcanization of rubber provided that the rubber has been produced from latex of similar concentration.*

C. If the reasons advanced above are accepted there remains the effect of the differential solubility of one or more of the non-caoutchouc constituents of latex in alcohol maintained at different pH values. That this difference in solubility exists is shown by the total solid content of the alcoholic serum liquids remaining after the coagulation of the samples described in exp. 222. See table IV.

TABLE IV.

Showing amount of total solids in the Alcohol—Serum Liquors.

Exp. Ref.	Optimum rate of vulcanization (minutes).	Total solids in serum (filtered).
		per cent.
222A. Acid cold	150	1.7
222B. „ hot	120	2.5
222C. „ cold	150	2.5
222D. „ hot	105	3.2
222E. „ cold	105	3.8
222F. „ hot	50	4.0

Note.—The serum from each coagulation was filtered. The clear filtrate was measured and an aliquot part was evaporated to dryness on the water bath the residue being dried to constant weight in the steam oven.

In all cases there was a change in colour and odour indicative of caramelisation.

* The influence of concentration of latex on the vulcanising properties of the resulting rubber has been made the subject of much investigation. Ref. publications by Whitby, De Vries, Eaton, Stevens and others.

A consideration of these figures in conjunction with the rates of vulcanization of the rubbers (which have been recorded again for convenience) gives rise to the supposition that the acceleration of vulcanization is due to the solution by the alcohol of a retarding agent which would normally be present in the coagulum and retained by the crepe rubber.

To examine this possibility the serum alcoholic liquor extracts were added to a control sample of standard fast curing crepe.* An aliquot portion of the liquor was dropped on to a known weight of crepe which was spread to dry on glass plates. The dry sample was mixed with the standard quantity of sulphur and vulcanized as usual. In a repeat test with the serum alcoholic extracts from 222A and 222F (the extremes) proportionate volumes of liquor were added to a definite weight of sulphur and the whole was dried off rapidly in a vacuum desiccater. The sulphur plus residue was then mixed with the necessary amount of fast curing crepe in the ordinary manner. The result are given in Table V.

TABLE V.

Rubber Sample Ref. No.	Serum Solids added from	Amount added on rubber.	Optimum rate of vulcanization minutes.	
222 Ap.	222 A. Acid cold	0.5	70	Serum alcoholic liquor added to rubber by spotting and air drying.
222 Bp.	222 B. „ hot	0.5	90	
222 Cp.	222 C. Neutral cold	0.5	70	
222 Dp.	222 D. „ hot	0.5	65	
222 Ep.	222 E. Alkaline cold	0.5	60	
222 Fp.	222 F. „ hot	0.5	60	
222 Rp.	Control	Nothing added	70	
222 XAp.	222 A. Acid cold	1.0	150	Serum alcohol liquor evaporated in vacuo with sulphur
222 XFp.	222 F. Alkaline cold	1.0	70	
222 X.	Control	Nothing added	80	

From these results it appear that the serum alcoholic liquors do not contain bodies which affect the rate of vulcanization of raw rubber to an extent sufficiently great to neutralize or overcome the slight influence of the small quantities of added acid or alkali.

This leads to the result that none of the explanations A.B. or C. suggested above account adequately for the facts observed as a result of the alcohol coagulations.

Possible accelerators in latex.—Further work was directed to the isolation of accelerating substances from raw rubbers which had been shown to possess divergent rates of vulcanization. In the next

* Crepe prepared from ordinary matured slab rubber. See F. M. S. Bull 27. "Vulcanizing" and "curing" being used synonymously in this article.

number of this Journal the separation by depolymerization of an insoluble portion will be is described. The material isolated from the following rubbers was examined for its influence on the vulcanization of normal crepe and "matured slab" crepe.

A. From rubber (represented by 222 K.L. above) derived from latex by "acid-alcohol" precipitation. Optimum rate of vulcanization $2\frac{1}{2}$ hours.

B. From rubber (represented by 222 M.N. above) derived from latex by "alkaline--alcohol" precipitation. Optimum rate of vulcanisation 75 minutes.

C. From a normal standard 1st latex crepe. Acid coagulation of latex according to standard estate practice. Optimum rate of vulcanization $2\frac{3}{4}$ hours.

D. From a matured slab rubber crepe.* Optimum rate of vulcanization 60 minutes.

Equal weights of these materials were machined with the rubber when making up mixes for 9 : 1 rubber sulphur vulcanizations. The "compounds" were then vulcanized side by with a control containing no added material. The results are given in Table VI.

TABLE VI.

Sample Ref. No.	Amount of Nitrogenous Insoluble Matter added to rubber.	Optimum time of Vulcanization (minutes).
287 Fc.	0.5% A.	180
287 Gc.	0.5% B.	180
287 Ac.	0.5% C.	180
287 Kc.	0.5% D.	180
287 Lc.	Nil. Control	180
287 Ms.	0.5% A.	60
287 Ns.	0.5% B.	60
287 Os.	0.5% C.	60
287 Ps.	0.5% D.	60
287 Rs.	Nil. Control	60

From which it appears that the nitrogenous insoluble matter isolated from raw rubber by depolymerization of an ordinary raw rubber does not possess accelerating properties when added to a 9 : 1 rubber-sulphur mix.

It may be argued here that the addition of nitrogenous residues to crepe is not the correct way to demonstrate their true effect on the vulcanization of rubber and sulphur. But in justification of the acceptance of deductions from the above results it must be borne in mind that the work is strictly comparative and it appeared reasonable to suppose that the parent rubber exhibited such marked differences on vulcanization that the derived insoluble products A.B.C.D. might be expected to reflect these differences when incorporated with a neutral rubber for vulcanization.

* For details of preparation see F.M.S. Bulletin No. 27, Eaton & Co., workers.

That the results are not in agreement with those published by Beadle and Stephens[3] or by Spence and Kratz[4] may be accounted for by the fact that in their work the effect of the insoluble matter was tried on the parent rubber and as evidence of the unsuitability of such a base for comparative trials the author desires to call attention to the influence of added starch and again litharge, a matter which will be referred to later in this article.

In order to gain further information on the accelerating effect of individual bodies contained in the residual serum of acid coagulated latex a separate investigation was conducted. The chemical aspects of that investigation will be described in the next number of this Journal, it remains to record here the vulcanization results obtained.

In experiment 263 air-dried naturally evaporated serum solids were added to fast and slow curing rubber from the same latex. A stock of latex was coagulated with acid and the coagulum drained from adherent serum liquor and divided into two portions; one of which was creped immediately and washed while the other was matured for 7 days before crepeing and washing.

The serum liquor was spread in a very thin layer in enamelled pans and allowed to dry in the air. After six weeks the liquor had dried to a black sticky substance which was collected and dried further in a vacuum desiccator. When suitable for weighing and mixing definite known weights of the serum solids were incorporated with the dry original rubber for vulcanization in a 9:1 rubber sulphur mix.

The following are typical of the results obtained :—

TABLE VII.

Sample.	Amount of substance added.	Optimum time of vulcanization (Minutes).
Original rubber Crepe	Nil	195
Crepe plus	1.25% serum solids	90
Original matured Crepe	Nil	95
Matured slab crepe plus	1.25 serum solids	75

In experiments 205 and 282 the effect was tried of the addition of various "individual" substances which had been isolated and examined as described in the forthcoming article on "Certain constituents of latex". It was found that the following bodies were without effect. Heat coagulable protein, magnesium pyrrol body coagulated by alcohol from serum, lecithin body, acetone soluble waxy substance.

In experiment 286 comparisons were made between the vulcanizing properties of ordinary crepe, matured slab crepe, the rubber substance obtained from spontaneously coagulated and evaporated latex (i.e. the rubber substance containing all serum solids), and the same rubbers to which extra serum solids* had been added prior to vulcanization.

* Prepared as already described under experiment 263.

The following table shows the results obtained :—

TABLE VIII.

Sample.	Description.	% Added Serum substance.	Optimum time of vulcanization (minutes).
286 Ac.	Ordinary Crepe	Nil	160
286 Asm.	Matured slab crepe	Nil	60
286 Aw.	Whole rubber by evaporation.	Nil	40
286 AcN.	Ordinary Crepe	2%	90
286 Asm.N.	Slab Crepe	2%	60
286 AWN.	Whole rubber by evaporation	2%	40

In experiment 296 an attempt was made to produce a rapidly vulcanizing rubber by the incorporation of serum solids which had been made to undergo decomposition. Serum derived from acid coagulated latex was first heated and the heat coagulable protein substance thus isolated, the residual liquor was then evaporated on a steam bath to a viscous brown syrup. These separate bodies were added to fresh water in the proportion of 10 grams per 100 cc. and set to decompose under aerobic conditions and under anaerobic conditions by inoculating with fresh latex. The contents of the dishes and tubes after 14 days were were made up to the original volume and were spotted on to pieces of a slow curing crepe rubber which were dried and then mixed with sulphur for vulcanization as usual, with the following results :—

TABLE IX.

Sample.	Description.	% Amount of material added to rubber plus sulphur.	Optimum time of vulcanization (minutes).
296 Ac.	Original protein substance	1.0%	135
„ Bc.	Undecomposed residue of serum solids	1.0%	135
„ Cc.	Protein decomposed aerobic	1.0%	135
„ Dc.	Serum residue decomposed aerobic	1.0%	120
„ Ec.	Protein decomposed anaerobic	1.0%	125
„ Fc.	Serum residue decomposed anaerobic	1.0%	135
„ Gc.	Control Crepe	Nothing added.	165
„ Hc.	Diphenyl guanidine	1.0%	15
„ BcR.	Similar to Bc.	2.0%	120
„ DcR.	Similar to Dc.	2.0%	90
„ FcR.	Similar to Fc.	2.0%	135

Experiment 299 shows the comparative results of vulcanizations made on ordinary crepe and matured slab crepe with the addition of decomposed serum solids unseparated and decomposed protein substance and decomposed "non-protein residue." A common stock of latex coagulated with acetic acid in the ordinary manner and the coagulum freed from adhering serum liquor, was divided into two portions one

of which was machined at once and washed thoroughly and the other matured for 7 days before machining and washing.

The serum liquor was divided into two portions X and Y. Y was treated thus—protein substance precipitated by warming to 60°C and maintaining until no further precipitate was obtained. The washed precipitate Yp was transferred to fresh water to which had been added 2 or 3 drops of latex and then allowed to decompose in very shallow layers in large open pans in which the surface of the liquid was freely exposed to the air. The residual liquor after the separation of the protein was evaporated as completely as possible on a steam bath. This substance called for convenience "serum residue" and labelled Ys was then added to latex inoculated water in the same proportion as Yp above and allowed to decompose under identical conditions.

The portion of serum liquor X was spread in a film approximately 0.5 m.m. thick on large glass plates which were set aside in the laboratory and protected from dust but to which air was allowed free access. Drying did not proceed to completion owing to the deliquescent nature of the serum solids, consequently after 7 days the required amount of material was added to the original dry raw rubber by rubbing a known weight of the rubber over the glass plates and reweighing the rubber at very short intervals until the required gain in weight due to adherent serum solids had been obtained.* The rubber plus serum solids was then sprinkled with the requisite weight of sulphur (1/9 weight of the dry rubber) and the whole was machined and mixed without any difficulty.

TABLE X.

Sample Ref. No.	Description.	Amount of material added % on the dry rubber.	Optimum time of vulcanization Minutes.
299Ac.	Standard crepe† machined at once.	Nil	135
299As.	Matured slab crepe machined after 7 days.	Nil	60
299AcX.	Crepe Ac. plus decomposed serum solids X.	3.0%	65
299AsX.	Matured crepe As. plus decomposed serum solids X.	3.0%	40
299AcYp.	Crepe Ac. plus decomposition products from protein substance Yp.	3.0%	90
299AsYp.	Matured crepe As. plus decomposition products from protein substance Yp.	3.0%	45
299AcYs.	Crepe Ac. plus decomposition products from Residue Ys.	3,0%	100
299AsYs.	Matured crepe As. plus decomposition products from residue Ys.	3.0%	60

* By proceeding slowly it was found an easy matter to make additions in a uniform manner and weighings were possible to 0.01 gramme.

† The latex used was strong consequently this crepe from an undiluted latex is not strictly "standard."

Consideration of the results recorded above, and many confirmatory tests which it is impossible on account of space to give here, leads to several conclusions, some of which have been recorded by previous investigators. For convenience they are tabulated here.

(*a*) Aerobic decomposition produces a more effective accelerating agent than anaerobic decomposition of protein substance or serum residue.

(*b*) The addition to raw rubber of serum solids or their decomposition products causes an accelerated vulcanization up to a point. However, it does not appear from these investigations that the rate of vulcanization can be accelerated beyond a well defined maximum by the addition of even large quantities of "natural" accelerators.

(*c*) The acceleration of vulcanization effected by the addition of a typical *artificial* organic accelerator, known also as a protein decomposition product, is very much greater than any acceleration observed for the addition of serum decomposition products.

(*d*) Rubber with the most extreme *naturally* accelerated vulcanization properties so far known is that produced by the intimate association of all the non-caoutchouc bodies of latex with the caoutchouc in evaported whole rubber.

In consequence of which, further work falls naturally into the following lines of investigation.

(i) Whether the accelerated vulcanizing properties of matured slab rubber can be reduced in any degree by continued washing of the rubber ?

(ii) The examination of differences between anaerobic and aerobic production of matured slab rubber.

(iii) Whether by increasing the proportion of non-nitrogenous substances in raw rubber the time of cure of matured slab rubber can be further reduced ?

(iv) The effect of evaporating latex under different conditions on the properties of the resulting whole rubber substance.

(i) The results obtained in this direction may be gathered from the following typical experiment.

A large piece of matured slab rubber* was cut into four portions and treated thus :—

A. Control portion machined 9 times to reduce the lump to crepe.†

Machine	1.	Three times.
,,	2.	do.
,,	3.	Twice.
,,	4.	Once.

* Dry coagulum. Grey on exterior and quite dry to handle but having a white interior containing 2.0% moisture.

† 4 machines were used.

The rolls of the machines were graded.

Machine 1.	having rolls set	¼" apart.
„ 2.	do.	1/8" apart.
„ 3.	do.	1/24" apart.
„ 4.	rolls set close for smooth crepeing.	

Copious washing was given on each machine.

B. Portion machined 12 times viz.

Machine 1.	three times.
„ 2.	do.
„ 3.	do.
„ 4.	do.

C. Portion machined 18 times as for B with additional 6 times in machine 4.

D. Portion machined 24 times as for C with additional 3 times in machine 3 and 3 times in machine 4.

E. Portion machined 36 times as for D with additional 6 times in machine 4.

F. Portion machined 3 times in No. 1 machine then soaked for 24 hours in water with a change of water at the 12th hour.

G. Portion machined twice in No. 1 machine and then soaked in water as for F.

H. Portion machined 3 times in No. 2 machine and then soaked in water as for F and G.

Extensive washing on each of the machines took place throughout the operations.

The creped rubber was then dried, mixed with sulphur and vulcanized.

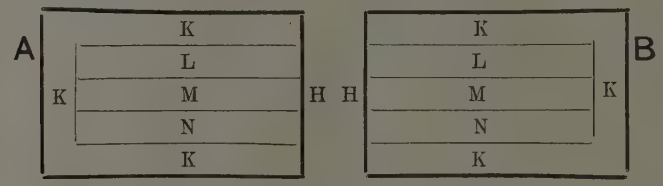
It was found that the optimum time of vulcanization—60 minutes was absolutely identical for all the samples A to H and the stress-strain curves of the vulcanized samples coincided for all times of cure proving absolutely that the rate of vulcanization of a raw rubber cannot be influenced by washing or machining and therefore that the accelerating agents are not removable by solution with water or by mechanical means.

(ii) The examination of crepe derived from different sections of a matured slab provides interesting observations on the enhanced vulcanizing properties of that type of rubber.

A bulk of latex was coagulated without the addition of acid, i.e. spontaneously, to form a large lump of coagulum. This was raised from the serum liquor, drained and divided into two portions A and B. These portions were allowed to mature on open racks in the air and were turned every alternate day. While the rubber was maturing the surface of A was kept absolutely free from the stinking alkaline slime

which normally forms, by rubbing over with a damp clean cloth every 24 hours. The surface of B was allowed to remain untouched, and the dirty slime developed in a normal manner.

The diagram shows how portions A and B were cut into sections. The rubber from each section in A was creped and washed very thoroughly while the rubber from B was creped uniformly with A but was not washed.



The results of one experiment are given in detail :—

TABLE XI.

Sample Ref. No.	Description of Sample.	Optimum time of vulcanization (minutes).
234 AH.	Outside section from clean slab	40
„ BH.	„ „ dirty slab	40
„ AK.	Exterior layer from clean slab	45
„ BK.	„ „ clean slab	45
„ AL.	Section L from clean slab	60
„ BL.	„ „ dirty slab	60
„ AM.	Section M from clean slab	60
„ BM.	„ „ dirty slab	60
„ AN.	Section N from clean slab	60
„ BN.	„ „ dirty slab	60

In a further experiment the mature rubber was derived from coagulum which had been obtained from *acid* coagulated latex. A large bulk of latex was coagulated with acetic acid according to standard estate practice and the coagulum obtained was divided into two portions and treated exactly the same as described under the previous experiment.

The results of vulcanizing the crepe samples are given :

TABLE XII.

Sample Ref. No.	Description of Sample.	Optimum time of vulcanization (minutes.)
236 AH.	Outside section of clean slab	40
„ BH.	„ dirty slab	40
„ AK.	Exterior layer from clean slab	40
„ BK.	„ dirty slab	40
„ AL.	Section L from clean slab	60
„ BL.	„ „ dirty slab	60
„ AM.	Section M from clean slab	60
„ BM.	„ M from dirty slab	60
„ AN.	„ N from clean slab	60
„ BN.	„ N from dirty slab	60

In a further experiment using latex containing less dry rubber per unit volume than that used for experiments 234 and 236 the coagulum derived by acid coagulation was matured complete in one piece. The matured slab was then divided into an exterior layer approx $\frac{1}{4}$" thick and an internal section, while a portion of the entire slab was kept intact. The three samples were then creped uniformly and the creped samples vulcanized.

TABLE XIII.

Sample Ref. No.	Description of sample.*	Optimum time of vulcanization (minutes.)
309 A.	Whole matured slab creped and washed.	45
309 B.	Exterior section creped and washed	20
309 C.	Interior section creped and washed	50
309 D.	Exterior section creped without any washing.	20
309 E.	Interior section creped without any washing.	45

The results recorded above, and many others, show that the exterior of matured rubber slabs possesses very different vulcanizing properties from the interior.

There are two possible explanations for this; firstly that the exterior contains a larger proportion of non-caoutchouc substances owing to surface exudation and evaporation, secondly that that the surface being in contact with the atmosphere is affected differently from the interior which is not in contact with the atmosphere. A chemical examination of the interior and exterior rubber does not indicate the presence in the rubber of an increased proportion of non-caoutchouc substances. The ash, the acetone extract and the Kjeldahl nitrogen are all within the normal limits for that type of rubber, and present technique does not allow the establishment of any significant differences. Again there is the direct evidence of experiment 234 and 236 in which comparisons were made between the rates of vulcanization of (B) slabrubber with which was incorporated all the slimy surface decomposition products and (A) slab rubber which was as far as possible deprived of the slimy surface decomposition products. The uniformity of the rates of vulcanization may be taken as additional proof of the correctness of the second explanation.

(iii) At this stage it is well to consider some results which were obtained by carrying investigations along the third line indicated above; to see if (and to what extent) an increased proportion of non-caoutchouc bodies, (nitrogenous and non-nitrogenous), would influence

* Practical assistance in the preparation of certain Samples has been given by Mr. Hooper of the F.M.S. Rubber Co. to whom thanks are due.

the vulcanizing properties of the matured rubber substance. One method of increasing non-caoutchouc bodies is to make an extract from one bulk of raw rubber and add the extract to a new bulk of rubber or replace the extract in fresh proportions.

A considerable volume of work has been done in that manner and in the case of the acetone extract there exists already a mass of published results to which reference can be made [5, 6]. In the present instance the results will be given of vulcanizations which were carried out on matured rubber, in which the non-caoutchouc constitutents had been increased by fractional coagulation of the original latex.

It has been established by repeated observation and recorded by many observers that a bulk of latex can be coagulated in such a manner that the rubber produced from the coagulum first formed differs from the rubber produced in later stages of coagulation.[7] The ash, acetone extract, and total N_2 (by Kjeldahl estimation) are all higher in the first coagulum than in rubber normally prepared. In one experiment, 40 gallons of undiluted latex* were treated with half the usual quantity of acetic acid. Three gallons were removed (E) set aside, and allowed to coagulate without further treatment. The remainder coagulated slowly with continuous stirring. After 30 mins. from the addition of the acid a small quantity of coagulum (A) was removed from the bulk of the otherwise completely liquid latex. Stirring was continued for a further 30 mins. and another small quantity of coagulum (B) was removed. Stirring and the subsequent removeal of coagulum at intervals of 30 mins. was proceeded with for sample (C) and (D) after which it was found that partial coagulation was not yielding quantities of coagulum suitable for subsequent treatment. An additional volume of acetic acid was added to the latex making the total quantity approximately equal to that normally employed† in latex containing 15% D.R.C. and the whole then allowed to coagulate. This coagulum (F) was obtained at the expiration of a further 90 minutes.

The fresh coagula A...B...F. were then divided into two portions Ac. Bc. Cc....Fc. which were washed thoroughly, creped and dried within 24 hours and portions As. Bs. Cs. Ds. Es. Fs. which were balled roughly to pieces of equal superficial area and set aside in the open air for 7 days to mature. Each matured slab was then cut into two sections, K, a layer cut as thin as possible from the interior of the slab, and M, the remainder, composing the interior of the slab. These sections were creped and washed very thoroughly in a uniform manner and the rubber was dried within 24 hours as for the samples creped previously.

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* Containing 40% D.R.C. i.e. 4 lbs. dry rubber per 1 gallon acetic acid coagulated latex.

† Normal coagulant is 3 ozs. of 5% acetic acid per 1 gallon of latex.

The results of comparative vulcanization are tabulated.

TABLE XIV.

Sample Ref. No.	Description.	Optimum time of vulcanization (Minutes.)	Optimum time of vulcanization of the dry crepe after extraction with cold acetone* (Minutes.)
267 Ac.	First fraction creped immediately.	165	135
,, Bc.	Second fraction creped immediately.	165	135
,, Cc.	Third fraction creped immediately.	165	135
,, Dc.	Fourth fraction creped immediately.	165	135
,, Ec.	Original bulk sample creped immediately.	210	180
,, Fc.	Residual latex bulk creped immediately.	210	180
,, ASK.	First fraction matured exterior creped.	40	65
,, BSK.	Second fraction matured exterior creped.	40	65
,, CSK.	Third fraction matured exterior creped.	40	65
,, DSK.	Fourth fraction matured exterior creped.	40	65
,, ESK.	Original bulk sample matured exterior creped.	55	75
,, FSK.	Residual latex matured exterior creped.	50	80
,, ASM.	First fraction matured interior creped.	40	75
,, BSM.	Second fraction matured interior creped.	40	75
,, CSM.	Third fraction matured interior creped.	40	75
,, DSM.	Fourth fraction matured interior creped.	40	75
,, ESM.	Original bulk sample matured interior creped.	90	95
,, FSM.	Residual latex matured interior creped.	90	95

The losses on extraction recorded, are particularly interesting for the matured rubber.

* These results are inserted here for convenience of comparison. The dry rubber crepe was extracted with cold, freshly redistilled, acetone in glass bottles in the dark for 10 days. The acetone was replaced every 24 hours. At the expiration of the 10 days the rubber was dried in a warm atmosphere—30°C, and weighed,

TABLE XV.

Sample.	Loss % by cold acetone extraction.	Sample.	Loss % by cold acetone extraction.
ASM. interior	3.0	ASK. exterior	1.8
BSM. do.	3.2	BSK. do.	1.3
CSM. do.	2.9	CSK. do.	1.8
DSM. do.	2.2	DSK. do.	1.9
ESM. do.	1.8	ESK. do.	1.5
FSM. do.	1.2	FSK. do.	1.8

The "acetone extracts" estimated by extraction with hot acetone in a glass Soxhlet extraction apparatus for 15 hours, drying and weighing the extract, were as follows:

Ac. 1st Fraction	Acetone extract % on dry rubber		...	2.2
Bc. 2nd	do.	do.	...	2.4
Cc. 3rd	do.	do.	...	2.5
Dc. 4th	do.	do.	...	2.6
Ec. Original		do.	...	1.8
Fc. Final residue		do.	...	1.5

From these results and others published it appears that the fractions, which contain the larger proportion of extractive matter, vulcanize more rapidly than the whole rubber or the residual rubber obtained from latex after the removal of those fractions; and maturation of the coagulum leads to an enhanced accelerated vulcanization for those coagula containing the larger proportion of non-caoutchouc bodies.

Further consideration of the comparative results for the rates of vulcanization of the dry creped from the interior and exterior sections of the matured slabs leads to the following conclusions:—

(*a*) The exterior of a matured slab from partially coagulated latex does not show a faster rate of vulcanization than the interior—though the bulk latex and residual latex do show considerable differences between the exterior and interior as previously recorded in experiments to 234 and 236.

(*b*) The differences in rates of vulcanization between the exterior sections of the fractions and the bulk latex coagula (after maturation) are comparatively small compared to the differences in rates of vulcanization displayed by the corresponding interior sections of the matured coagula and the large differences between the corresponding coagula creped before maturation.

(*c*) Extraction with cold acetone leads to a corresponding accelerated rate of vulcanization for all the samples Ac.—Fc. but not so for the same coagula after maturation. Extraction of the matured dry rubber leads to a slower vulcanization for all the samples, though the retardation is practically negligible for the samples Esm.

and Fsm. and a pronounced difference appears between the rates of vulcanization of the exterior and interior of the partially coagulated slabs.

The striking fact is that these alterations in rates of vulcanization do not appear to be related to the losses in weight of the raw rubbers nor can they be accounted for by the values for the acetone extracts of the crepe samples.

An examination of the extracts obtained by the cold extractions has not led to the conclusion that the extracts from samples Ac. ¬Fc. contain accelerating substances but the reverse, and it has already been stated that two separate substances isolated from latex as acetone soluble bodies are without influence on the rate of vulcanization of raw rubber. On the other hand the result of earlier work by Beadle and Stevens Van Heurn, and recently by Stevens[10] tends to lead to the conclusion that acetone extraction may not always produce uniform alterations even with a uniform type of rubber. It it significant that Stevens has recorded as a result of his later work, the conclusion that "the correlation between the amount of acetone extract and rate of vulcanization is not directly connected with the presence of putrefaction bases, as acetone extraction which should remove the bases may result in a more rapid vulcanizing rubber."

The work detailed in this section again serves to show that the increase of water soluble non-caoutchouc constituents or their putrefactive decomposition products does not entirely account for the accelerated rate of vulcanization of matured rubber, whereas the difference between the exterior and interior of slab rubber is highly significant of a factor in maturation which has hitherto been unconsidered.

(iv) *The rate of vulcanization of whole rubber.*—Leaving further discussion of the results recorded above and their bearing on established views, it is necessary to refer back to the fourth line of investigation mentioned earlier.

An investigation of the vulcanizing properties of the whole rubber produced by evaporating latex under different conditions was carried out. Unfortunately, circumstances have precluded the possibility of examining more than the merest fringe of the problem but enough has been achieved to lead the writer to the conclusion that the results or significant and they appear to be relevant to the present issue.

"Latex, undiluted, aud treated with the minimum delay after flowing from the tree may be evaporated to a uniform transparent film of rubber material which on testing under the standard conditions exhibits a comparatively rapid rate of vulcanization in a 9: 1 rubber sulphur mix, being normally about 60 minutes, which is practically the same as that for matured slab rubber under the same conditions. There can however be no question of the development of *putrefactive* bases in the evaporated whole rubber because the time elapsing from tapping the latex to vulcanization of the rubber has on serveral occasions been no longer than 3 hours and an examination of the latex and the derived rubber has been controlled by bacteriological examinations which practically precluded the possibility of putrefactive changes.

The rate of vulcanization of this evaporated whole rubber shows some striking variations on certain days, although control samples of acid coagulated creped and matured slab rubber from the same latex does not reflect these variations; it does not appear possible at the moment to correlate the variations to differences in the chemical constants of the latex examined. It is necessary to add in this connection that the work so far carried out has not been more than superficial and it will be obvious to investigators familiar with the normal daily variations in latex that any results must be recorded over a long period before they can be interpreted.

Apart from these variations which may for the present be regarded as inherent to the latex there are variations, in the vulcanizing properties of the whole rubber, which are due to external influences during preparation of the rubber substance.

These may be illustrated by quoting the results of actual experiments.

TABLE XVI.

Sample Ref. No.	Description of Method of Preparation.	Optimum time of vulcanization (minutes).
305 X.	Latex spread on glass plates. Drying complete in 20 mins. at 45°C* in drying chamber.	60
305 Y.	Same latex transferred to dark room on collection spread on glass plate in dark room under a fan. Drying complete in 3 hours at 25°C.	120
305 X.	Same latex spread on glass plates in laboratory in bright diffuse light. Drying complete in 3 hours at 30°C. Rubber film allowed to remain exposed for 24 hours before mixing with sulphur.	120
305 Ac.	Same latex coagulated with acid, coagulum raised and creped and the crepe dried in the ordinary manner.	150
305 W.	Same latex through which air had been blown for 60 minutes; then coagulated with acid, coagulum raised, creped and the crepe dried in the ordinary manner.	150
305 AS.	Same latex coagulated, coagulum raised and matured for 7 days, then creped etc. as for 305Ac.	
306 A.	Latex spread on glass plate in hot chamber Drying complete in 15 minutes at 45°C.	70
306 B.	Same latex spread on glass plates in dark room under fan. Drying complete in 3 hours at 25°C.	120

* The glass plates 92cms. x 23cms. area were used as shelves in a drying chamber; 25ccs. of latex were spread on each plate. The interior of the chamber was heated by steam pipes at the bottom and the air circulated by a suction fan in the top. Temperature was controlled by a thermometer projecting into the centre of the chamber.

Sample Ref. No.	Description of Method of Preparation.	Optimum vulcanization time of (minutes).
306 C.	Same latex spread on glass plates in laboratory under fan. Drying complete in 2 hours.	120
306 D.	Same latex treated exactly as 306A. but kept in hot chamber for a further 24 hours.	90
306 E.	Latex spread on glass plates in hot chamber. Drying complete in 15 minutes at 45°C.	90
306 F.	Same latex spread on glass plates in dark room under fan. Drying complete in 3 hours. Mixing of rubber with sulphur after 24 hours.	120
306 G.	Same latex spread on glass plates under dark room exactly as 306F. above but maintained in dark room for a further 48 hours.	240

A very marked difference in appearance is found to exist between the rubber prepared by evaporating latex in the warm chamber and that prepared by slower evaporation in the laboratory and the dark room. The former rubber 305X., 306A., 306E. and other samples not mentioned here are absolutely transparent, almost colourless, and possessed of a good elasticity in the raw state. The latter rubber 305Y., 306F., 306G., etc. are opaque samples giving a brown rubber lump and is almost devoid of elasticity in the raw state.

The tensile strength of all the samples after vulcanization was normal and did not exhibit any material differences.

These results taken in conjunction with those published by previous investigators[9] on evaporated latex are, in the writer's opinion significant.

It has been established by the work of Weber, Herbst, Peachey and others that caoutchouc forms a definite series of oxidation products and that these appear to be additive compounds of the type $(C_{10}\ H_{10})$. Further investigations have shown that the oxidation of caoutchouc is an autoxidation and the curves connecting oxygen absorption with time, obtained by Peachey, are characteristic of an autocatalytic process[11]. This view further developed by Kirchof suggests the formation of an unstable peroxide as an intermediary in the oxidation of caoutchouc.

If such views are considered in conjunction with the results of certain work by Kerbosch[12] on the susceptibility to oxidation of certain samples of rubber, before and after removal of the latex serum substances, and under the influence of added albumin it will be seen that the method of preparation of rubber may lead to important variations in the composition of the material, which will be unconnected with putrefactive changes.

This article has been written with the object of demonstrating what appears to the author to be a significant connection between the oxidation of caoutchouc and the rate of vulcanization of raw rubber.

There has been little doubt on the part of others that the nitrogenous constituents of the non-caoutchouc substances accounted for a part only of the vulcanization properties of raw rubber[16]. The present work justifies and confirms those views and in addition brings out results which seem to prove the importance of oxygen in contact with the caoutchouc itself.

The presence of a peroxidase in Hevea latex has been recorded[13] and it has been understood that the enzyme is brought down with the rubber when latex is coagulated. The extraction of a peroxidase from the surface of "Fine Hard Para" rubber was recorded by Spence over fourteen years ago. The distinctive colour of the fractions obtained in the partial coagulation of latex by acid has been attributed to the increased quantity of an unknown yellow body which is connected with the presence of oxidation products and an oxydase.

The facts recorded under experiments 234, 236 and 309 in which the exterior of a matured slab was shown to possess more rapid vulcanizing properties than the interior of a matured slab, the fact that the rate of vulcanization is not affected by washing with water, and also the facts recorded under experiment 267 appear to justify the conclusion that the rate of vulcanization of raw rubber is largely influenced by the presence in the rubber substance of caoutchouc oxidation products which may be first formed through the agency of a peroxidase in latex.

It is suggested that the initial stage in the oxidation whether through a peroxidase or an autocatalytic process gives rise to the formation of a labile peroxide, similar to that formed from terpene substances and studied more particularly in the case of oil of turpentin. The presence of the labile peroxide will in an atmosphere of sulphur vapour cause accelerated vulcanization but in an atmosphere of oxygen, or, in air further oxidation takes place with the formation of oxygen saturation products of caoutchouc. These are the natural products of ageing of raw rubber and consequently the rate of vulcanization of aged sample will differ materially from the originals.

In some cases ageing may lead to an acceleration of vulcanization owing to the formation of peroxide, but in other cases in which the maximum proportion of peroxide has been formed and the original sample was a rapid vulcanizing rubber, ageing will cause a retardation.

There is so far no definite proof of the enhanced vulcanizing properties of caoutchouc at certain stages of oxidation and work in that direction is be set with difficulties which will only be overcome slowly, but an opening has been made on the examination of films of latex-evaporated rubber and their properties after vulcanization by a cold process. In a subsequent publication it is proposed to give a description of the apparatus and method of work and a resume of the initial results.

In conclusion the author desires to acknowledge his indebtedness to the help which has been given by Mr. W. N. C. Belgrave, Plant Physiologist and to record the fact that the entire work has been carried on as a result of discussion with him.

Summary.

The coagulation of latex by dropping into alcohol of variable hydrogen ion concentrations produces samples of rubber which exhibit significant variation variations in rate of vulcanization.

These variations are not accounted for by simple chemical or physical explanations based on previously accepted views regarding the variations occuring in raw rubber.

Attempts to account for the facts observed and considerations of the properties of depolymerized rubber residues, various serum substances and their decomposition products, gives rise to the supposition that it is the caoutchouc of raw rubber which is most intimately concerned in the changes taking place when coagulum is "matured."

Consideration of the important differences in "maturation" brought about by the air and the effect of acetone extractions leads to the further supposition that the caoutchouc forms oxidation products which exert a profound effect on its vulcanization properties.*

Previous work on the oxidation of films of rubber and the author's present results on the vulcanization of latex evaporated rubber tend to confirm these suppositions and it is suggested that the ageing of raw rubber is a natural sequence of "maturation" and its effects.

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* The presence of a peroxidase in latex and its possible effect on a wet coagulum may account for the differences observed between the mature rubber from hot and cold alcohol-coagulated latex.

PRELIMINARY REPORT ON THE DISTRIBUTION BY THE INSPECTION DIVISION IN PERAK NORTH OF SELECTED PURE STRAINS OF PADI.

By F. Birkinshaw.

AS the result of the work of several years by the Economic Botany Division there are in existance at the Titi Serong Rice Experiment Station isolated pure strains of padi capable of producing higher yields than the unselected varieties of the country. The aim now is to get these high yielding pure strains generally known and grown by cultivators and the following is an account of the results achieved in this direction during the past two seasons by the Inspection Division in Perak North. This division of the State comprises the Districts of Krian, Larut (including the sub-districts of Matang and Selama) Kuala Kangsar and Upper Perak.

Reasons why proper control of distribution is necessary.

The introduction of improved strains of padi to Malay cultivators is not such a simple matter as it may appear to be at first sight. The despatch of a quantity of selected pure line seed to a number of centres for distribution to cultivators would be no guarantee of improved yields throughout the country for the following reasons :—

Firstly. The cultivator's prejudice in favour of local varieties which he has habitually grown. In each locality the Malay cultivators have a strong predilection for the varieties of padi which have been grown in the locality for a number of years. This partiality is justified to a large extent, for it is certainly true that the varieties grown in a particular locality are amongst those most suited to local conditions of soil, irrigation and methods of cultivation. The result of an uncontrolled broad-cast distribution would, therefore, be either that the distributed seed would be used for food, or, if sown, would be planted on a small portion of the worst land in the locality concerned, the best land being reserved for the local varieties in which the cultivators place their faith. Under such conditions the selected pure strain would probably give a lower yield than the local varieties. The cultivator would have proved to his own satisfaction the superiority of his local varieties and thereafter would refuse to plant any other.

Secondly. The necessity to select for distribution a strain of a variety suitable for its intended environment. From this standpoint it may be pointed out that conditions vary, not only in different parts of a District but also in different fields in a locality and sometimes even in different portions of a field itself. Less efficient irrigation on one portion of a field as compared with other portions may make it advisable to distribute two dissimilar varieties or strains for the same field.

Thirdly. The necessity to guard against the deterioration of pure strains through admixture with unselected seed.

Fourthly. The importance of determining and recording comparative yields between distributed strains and local varieties. This not only helps to determine the suitability for its environment of the pure strain, but also may suggest the advisability of attempting the isolation of a high yielding pure strain from one or more of the local varieties.

Fifthly. The necessity of provision for making comparative tests of pure strains in a number of localities in order to determine the most suitable strain for each locality.

Distribution Scheme.

To meet the above points preliminary distributions were made only to certain reliable cultivators who might be expected to give the strain distributed to them a fair trial. At Talang, Kuala Kangsar, a test station was established last year where suitability of certain strains for the locality was tested. Another test station was laid out at Selinsing by the Penghulu. In several other localities where cultivators have gained sufficient confidence in the selected strains for the purpose, practical test stations have been arranged for the coming season by supplying a number of strains to the same number of cultivators, one strain to each. As far as possible measurements of comparative yields between selected strains and local varieties have been made and recorded. Inspections of the selected strains in the field have been carried out in so far as time would allow.

Distributions made for season 1921-22.

In Feburary 1921 the District Officer, Kuala Kangsar, Perak, applied to the Department for five bags of selected seed for distribution in his district. This amount was later reduced to 2 bags each containing about 25 gantangs, one of Seraup No. I. the other of Radin No. I. Partly by reason of the partiality of cultivators for their local varieties as mentioned earlier, and partly because of a prejudice created against Government-introduced seed owing to badly controlled distribution during the food shortage period in the later years of the war, the demand for the selected seed was so small that the District Officer distributed only 20 gantangs as follows:—

To Whom.	Seraup No. I.	Radin No. I.
Penghulu, Chegar Galah	2 Gantangs	2 Gantangs
„ Senggang.	4 „	4 „
Assistant Penghulu, Pulau Kimiri	2 „	2 „
Penghulu, Kampong Buaia	2 „	2 „

It was agreed that the Inspection Staff should, as far as other work permitted, keep in touch with the seed supplied. In every case the padi grew well and returns were good but most of the cultivators stated that the yields were no better than those obtained from local varieties. In these cases seed was not kept for sowing in the season following. There were exceptions in the case of localities in the mukims of Kampong Buaia, Chegar Galah and Pulau Kimiri where redistributions of seed were made.

Re-distribution by cultivators for 1922-23 season.

1. Kampong Buaia mukim.

(a) Bendang Ulu Papan. The Ketua here was supplied with 1 gantang of Seraup No. I seed for 1921-22 season. For 1922-23

season he distributed over 90 gantangs or sufficient to plant about 25 acres.

(b) Kampong Lalang. The Ketua grew 1 gantang of Seraup No. I in 1921-22 season. Of the seed obtained he re-distributed over 35 gantangs or sufficient to plant about 10 acres. Comparative yields for these two localities will be found tabulated below.

2. Pulau Kimiri mukim. Redistributions were made here of 13 gantangs of each.

3. Chegor-Galah mukim. A little redistribution was made in the locality of Jawang

TABLE I.

Record of yields 1922-23 season from pure strains grown from redistributions made by cultivators who grew the seed originally distributed in 1921-22 seasons, with some comparative local variety yields.

(1) Distributed by Haji Ishak, Ketua, Bendang Ulu Papan.

Locality grown.	Pure strain or local variety.	Maturation period.	Yield per acre in gantangs.
Bendang Ulu Papan.	Seraup No. I.	8 months	650 pure strains
,,	,,	,,	570 ,,
,,	Padi Bemban	,,	700 } Local va-
,,	Padi Chantek	7½ ,,	590 } rieties
Bendang Gapis.	Seraup No. I.	8 ,,	425 } Pure
Bendong Ulu Papan Ekor.	,,	,,	490 } Strains
,,	Padi Chantek	7½ ,,	506 Local variety

(2) Distributed by Anjang Rahim, Ketua, Kampong Lalang.

Kampong Lalang	Seraup No. I.	8 months	880 Pure strains
,,	,,	,,	690 ,,
,,	,,	,,	720 ,,
,,	,,	,,	775 ,,
,,	,,	,,	740 ,,
Paya Panjang	,,	,,	750 ,,
Kampong Stor	,,	,,	650 ,,
Bendang Pulai	,,	,,	647 ,,
,,	Padi Machang	,,	575 Local variety

No measurements were taken of the padi redistributed in Pulau Kimiri mukim.

At Jawang the pure strains grew remarkably well, but a little before harvest swarms of rats made their appearance and did so much damage that it was decided no reliable measurement of yield was possible.

TABLE 2.

Record of yields 1922-23 season from pure strains distributed, with some comparative local variety yields.

District of Kuala Kangsar.

Mukim.	Locality.	Pure strain or local variety.	Maturation period.	Yield per acre in gantangs.
Kampong Buaia	Kg. Lalang	Radin No. 1	7 months	570 Pure strains.
,,	,,	,,	,,	480 ,,
,,	Paya Panjang	Radin No. 7	7½ months	410 ,,
,,	Kembai Batu	,,	,,	620 ,,
,,	Kg. Lalang	Radin Che Mah	7 months	560 Local varieties.
,,	,,	Padi Bujang Berinei	,,	600 ,,
,,	Kg. Stor	Radin Che Mah	,,	485 ,,
Lubok Merbau	Langkor	Seraup Kechil No. 20	8 months	660 Pure strains.
,,	Kubang	,,	,,	610 ,,
,,	Tanah Merah	Radin No. 7	7½ months	600 ,,

District of Larut and Matang.

Simpang	...	Seraup No. 36	8 months	350 Pure strains.
,,	...	Muda Che Alli	,,	355 Local variety.
Bukit Gantang	B. Jelutong	Seraup No. 36	,,	490 Pure strains.
,,	,,	Radin No. 11	7 months	485 ,,
,,	,,	Padi Puteh	,,	460 Local varieties.
,,	,,	,,	,,	480 ,,
Tupai	Larut Ulu	Radin No. 1	,,	225 Pure strains.
,,	,,	,, 13	,,	330 ,,
,,	Larut Tengah	,, 1	,,	560 ,,
,,	,,	Padi Chantek	7½ months	415 Local variety.
,,	Parit Mentri	Radin No. 13	7 months	460 Pure strain.
,,	,,	Padi Pahit	7½ months	540 Local variety.

TABLE 3.

Record of yields at Talang Test Stations 1922-23.

Pure strains grown in quantity.

Pure strain or local variety.	Yield per acre.	Mean yield for each strain.
Radin No. 1	460	455 gantangs per acre.
,,	470	
,,	380	
,,	495	
,,	450	
,,	480	
Radin No. 2	580	465 gantangs per acre.
,,	375	
,,	430	
,,	360	
,,	490	
,,	550	
Radin No. 7	450	475 gantangs per acre.
,,	450	
,,	430	
,,	455	
,,	600	

Comparative tests of pure strains grown by the Kotua.

Radin No. 1	460	
,, 2	580	
,, 3	380	
,, 7	450	
,, 13	600	

Local Varieties.

Radin Puteh	340	470 gantangs per acre.
,,	440	
,,	510	
,,	585	

Pure strain pulut varieties.	Local pulut.
Hitam F.S. 644-490 gantangs per acre.	Pulut Pipit—595 gantangs per acre.
Cheakbong F.S. 670-600 gantangs per acre.	Pulut Serai—580 gantangs per acre.

Remarks on above tables.

I propose to preface my remarks by some observations upon the pure strains and upon the introduction of such to new localities.

The Pure Strains.—These have been isolated from amongst a large number of varieties selected from throughout the Federated Malay States. When grown at Titi Serong those chosen for distribution have shown on consecutive comparative tests with local varieties a higher yield of from 20% to 25%. Other advantages claimed for the pure strains are uniformity of ripening within each strain and a higher weight per unit volume as compared with local varieties. Hence the claim that production throughout the country will be increased by replacing present varieties by our pure strains has reason to support it.

Introduction to fresh localities.—Padi, in common with many other crops belonging to the same Natural Order, is very susceptible to changes in environment. This point has a bearing on the distribution of pure strains of padi in that it gives some ground for assuming the possibility that a pure strain introduced to a fresh locality may be capable of higher yields after being grown for several consecutive years in the locality than upon its first introduction. In other words the susceptibility to environment also suggests capability of adaptation to environment. This shows the impossibility of judging results from one or two years records of comparative yields and emphasises again the need for test stations and systematic control of distribution.

Table 1.—This table is concerned with one pure strain, Seraup Kechil No. 1. At Bendang Ulu Papan the local variety Bemban yielded 700 gantangs per acre as against 650 gantangs from Seraup Kechil No. 1. The local variety is a Seraup Kechil. It is a striking example of a variety being eminently adapted to local conditions. Another pure strain of Seraup Kechil will be introduced to this locality next season for further comparison. It is proposed also to raise a pure strain from the local Bemban for comparison with the existing strains at Titi Serong. At Kampong Lalang (including other similar bendangs in the locality) the mean of the measurements of yield from eight separate plots was over 730 gantangs per acre as against a yield of 575 gantangs from the best local variety "Machang." In this instance the yield of the pure strain may be safely assumed to be over 25% higher than the best local yield, for the "Machang" may be relied upon to be the very best in the locality, having been selected for comparison by the cultivators themselves.

The pure strain used here and at Bendang Ulu Papan is not our best strain of the variety.

Two better strains will be introduced next season at both localities. The bendangs of Bendang Ulu Papan Ekor and Bendang Gapis are not so suitable for Seraup as the remainder of the bendangs mentioned and trials with pure strains of other varieties will be made next season.

Table 2.—All the pure strains mentioned here have been grown for only one season in the localities mentioned. As a result of more thorough knowledge of the bendangs and requirements for the various localities, pure strains of Seraup and Padi Pahit will be introduced next season in several instances to replace or supplement varieties of Radin. This applies especially to some localities in Larut District. Trials with Radin No. 1 will be discontinued or almost so. Radin No. 7, 11 and 13 will be continued within certain localities and Radin No. 4 introduced to certain bendangs. Trials with Seraup Kechil No. 20 will be continued at Lubok Merbau in Kuala Kangsar District. Little more can be said regarding table 2 excepting that it will provide a record for comparison with next season's records.

Table 3.—As a result of this year's test it has been decided to utilise Radin No. 13 for the major portion of Talang test station. Further trials will be made with all the strains used this year and Radin No. 4 and 11 will be introduced. Seraup No. 36 and 52 will be utilised to plant up a low lying part of the station. With regard to Radin Puteh which was used for comparison, it was found, unfortunately only when too late, that this variety is the progeny of a selected padi introduced to the locality some years ago by the Economic Botanist who used it to plant up the station in connection with experiments undertaken by him. It is therefore not really a local variety but one of the Titi Serong pure strains.

The average yield per acre of between 450 and 475 gantangs obtained from the station is much higher than the local yields of the year for places in the same mukim.

Talang Test Station.

The portion of the station cultivated this year was about 12 acres in extent. The padi was grown by local Malays who received land free of rent on condition that pure strain seed would be grown and that cultivation would be reasonably well attended to. The produce, excepting what is needed for seed in the following season, becoming the property of the cultivator. This method proved sufficiently satisfactory to warrant its continuance here and adoption for other test stations which may be established. Preliminary cultivation was not so good this year as it should have been as over most of the area only the "Tajak" was used. For the type of land something more is needed and the cultivators were informed that ploughing and rolling must be done next season, being given the option to promise to do so or to give up the land. All save one complied.

Reference to the table 3 for yields of Radin No. 2 will illustrate the effects of cultivation on yield. In the two plots yielding 580 gantangs and 550 gantangs per acre respectively, both the "Tajak" and "Golek" were used, whereas on the other four plots giving a mean of just over 400 gantangs per acre the "Tajak" only was used. The major portion of the bendang was planted with Radin Nos. 1, 2 and 7. The other strains shown in Table 3 were planted in small plots on an uniform piece of land for comparative trials. The few pure strains of padi pulut were introduced in response to a request by

the cultivators that each should be allowed to grow a small quantity of pulut padi.

Concluding remarks.

Pressure of other work and limitation of staff has not permitted the carrying through of all that was intended. In many instances no measurements of yields were made and in others no records of local yields for comparison were obtained. A little progress however has been made and a gradual tendency towards better execution is hoped with increased experience by the staff and increased confidence of the cultivators in both the staff and in the pure strains of padi advocated by us.

The above progress report was written when I was Assistant Agricultural Inspector, Perak North. Its publication was postponed to allow of consideration of the advisability of including in the one report results obtained from the distribution of pure strains of padi by the whole of the Inspection Staff throughout the Federated Malay States and Straits Settlements.

After careful consideration it appeared to me when acting Chief Agricultural Inspector more desirable to publish the original report, as remarks therein on the Distribution Scheme, need for methodical control and the careful test and selection of suitable pure strains for particular localities apply equally throughout Malaya. The inclusion of all results would entail more voluminous tables without any corresponding material addition to the text. Below is a brief summary of pure strain distribution by the Inspection Division in other districts.

Perak South.—Comparative trials were made at Gopeng with four pure strains of Radin and two pure strains of Seraup Kechil.

Pahang East.—At Paya Kelat Rendang, Pekan, comparative trials were made with pure strains of Radin and Pahit.

Province Wellesley.—In the North District seed was distributed in ten localities to 17 cultivators, the pure strains used being Radin Nos. 1, 7 and 13, Seraup Besar 15, and Seraup Kechil 36. In the Central District pure strain seed of Radin 1 and 2 was issued to five cultivators. In every case yields were obtained and recorded as well as yields of local varieties for comparison.

A test station was established last season at Pertang To' Jaya in which comparative tests were made of eight pure strains, Radin Nos. 7 and 13, Seraup Besar 15 and 617, Seraup Kechil 36, 52 and 371. The best yields obtained were from Radin 7, Seraup Kechil 52 and Seraup Besar 16 which yielded at the rate of 590, 520 and 512 gantangs respectively.

Malacca.—Comprehensive trials were arranged in this settlement in 31 mukims representing the main padi growing areas, the following pure strains being used:—

Radin 1, 7, 11 and 13, Seraup Kechil 52,371, Pahit 1 and 4. Records of yields of the pure strains were obtained from 57 plots situated in various parts of the Settlement and of local varieties for comparison from 35 plots. The yields obtained in many instances are very promising.

General Remarks.—In a great many instances the yields obtained from pure strain padis, even in the first trial, are such as to warrant the belief that each such strain will become extensively planted after a few seasons in the locality to which it has been introduced and the result will be an increased yield generally for the locality. In other instances there are indications that, although the pure strain yields were not higher than that of local varieties, the pure strain used is capable of adaptation to its new environment and will give increased yields after a few seasons. The yield records as a whole coupled with the experience gained last season, are of great use in helping the Assistant Agricultural Inspector concerned to form an opinion as to the strain most likely to prove suitable for any particular locality.

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LONDON MARKET PRICE LIST, 3rd QUARTER 1923.

OIL SEEDS.

Castor (Bombay)	-	£19 per ton.
Coconut (Desiccated)	-	37/- per cwt.
Copra (Ceylon)	-	£26 per ton.
,, (Straits)	-	£24 ,,
Cotton (Egyptian)	-	£10—£11 per ton.
,, (Bombay)	-	£9—£9.5 ,,
Croton	-	27/6—32/6 per cwt.
Gingelly (Chinese)	-	£23 per ton.
,, (Bombay)	-	£22 ,,
Groundnuts (Gambia) undecorticated	-	£17.10 per ton.
,, (Chinese) decorticated	-	£21.10 ,,
Linseed (Bombay)	-	£19.7.6 ,,
,, (Plate)	-	£18.5 ,,
Palm Kernels (West Africa)	-	£16.10 ,,

OILS.

Castor (Madras)	-	49/- per cwt.
,, (Pharmaceutical)	-	61/- ,,
,, (1st pressing)	-	56/- ,,
,, (2nd pressing)	-	55/- ,,
Coconut (Cochin)	-	47/- ,,
,, (Ceylon)	-	38/3 ,,
Cotton seed (Egyptian, crude)	-	39/6 ,,
,, (Bombay)	-	37/- ,,
Groundnut (Oriental, crude)	-	46/- ,,
,, (English)	-	43/- ,,
Linseed (Calcutta)	-	41/- ,,
,, (Plate)	-	40/6 ,,
Palm (Lagos)	-	£32.10 per ton.
,, (Congo)	-	£28 ,,
Palm kernel	-	35/6 per cwt.

OIL CAKES.

Coconut	-	£7—£8 per ton.
Groundnut (Semi-decorticated)	-	£10 ,,
,, (undecorticated)	-	£8 ,,
Linseed	-	£10 ,,
Palm kernel	-	£5 ,,

ESSENTIAL OILS.

Cajeput	-	3/3—3/6 per lb.
Camphor (Chinese, crude)	-	3/- ,,
,, (Japanese, refined)	-	3/6 ,,
,, (oil)	-	75/-—80/- per cwt.
Cinnamon (Ceylon, leaf)	-	4d. per oz.
Citronella (Ceylon)	-	3/6 per lb.
,, (Java)	-	4/2 ,,
Clove	-	7/- ,,

OIL CAKES—*Contd.*

Lemon grass (Cochin)	-	2½d. per oz.
Lime (West Indian, expressed)	-	7/-—7/6 per lb.
„ (West Indian, distilled)	-	3/6—3/9 „
Nutmeg	-	4/6 „
Patchouli (Penang)	-	27/-—30/- „
Vetiver (Bourbon)	-	21/6 „
Ylang Ylang	-	15/-—16/- „

SPICES.

Capsicums (East Indian)	-	40/-—45/- per cwt.
„ (Nyassaland)	-	60/-—65/- „
Chillies (Zanzibar)	-	65/-—75/- „
„ (Nyassaland)	-	85/-—95/- „
„ (Japan)	-	125/- „
Cinnamon (Ceylon)	-	7½d.—10½d. per lb.
Cloves (Zanzibar)	-	1/1 „
„ (Penang)	-	2/6—3/- „
Ginger (Japanese, Cochin)	-	70/-—80/- per cwt.
„ (Jamaica)	-	180/-—200/- per cwt.
Mace (Bombay & Penang)	-	1/4—1/8 per lb.
Nutmegs (Singapore & Penang)		
110's	-	10½d „
80's	-	1/1½d. „
64's—57's	-	1/7—2/- „
Pepper (Singapore, black)	-	4d. „
„ (Singapore, white)	-	8d. „
Turmeric	-	45/- —70/- per cwt.

DRUGS.

Areca	-	35/- —50/- per cwt.
Cocaine (hydrochloride)	-	15/- per oz.
Ipecacuanha (Matto Grosso)	-	8/- per lb.
Quinine Sulphate	-	2/8 per oz.

NATURAL DYESTUFFS & EXTRACTS.

Annatto (seed)	-	1/6d. per lb.
Cutch (Borneo, solid)	-	40/-—45/- per cwt.
Gambier (block)	-	60/- „
„ (cubes)	-	62/6—65/- „

GUMS.

Damar (Singapore)	-	60/-—250/- „
„ (Batavia)	-	100/-—120/- „
Dragon's blood (reeds)	-	£18—£22 per cwt.
„ „ (lump)	-	£11—£25 „

FIBRES.

Cotton (F. M. American)	-	11½d. per lb.
„ Egyptian Sakellaridis)	-	10/10d. „
Hemp (African sisal)	-	£30 per ton
„ (Manila, J. Grade)	-	£30 „
„ (New Zealand)	-	£30 „
Kapok (Java)	-	1/2½ per lb.
„ Indian)	-	10½d „

FOODSTUFFS.

Cocoa (Ceylon, plantation)	-	60/-—85/- per cwt.
Coffee (East India)	-	85/-—120/- „
Sago (pearl)	-	24/-—30/- „
„ (flour)	-	16/-—17/- „
Sugar (White Java)	-	49/6—50/- „
Tapioca (Penang, flake)	-	3¼d.—3⅜d. per lb.
„ (Penang, flour)	-	18/-—22/- per cwt.

MISCELLANEOUS CHEMICALS.

Acetic acid (glacial)	-	£71 per ton.
„ (80% comml.)	-	£50 „
Acetone (pure)	-	£130—£135 per ton.
Ammonia (.880)	-	£32—£34 „
Calcium acetate (grey)	-	£22—£23 „
Citric acid	-	1/6 per lb.
Creosote	-	9d. per gallon.
Formalin (40% vol.)	-	£97—£98 per ton.
Lime juice (raw)	-	2/9 per gallon.
„ (conc.)	-	£22 per basis.[1]
Sodium bisulphite	-	£19—£20.10 per ton.
„ sulphite (anhydrous)	-	£27.10—£29 „

Received for publication September 26th 1923.

1 Basis=108 gallons, 64 ozs. Citric acid per gallon.

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